

Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/GB05/000134

International filing date: 20 January 2005 (20.01.2005)

Document type: Certified copy of priority document

Document details: Country/Office: GB
Number: 0401561.6
Filing date: 24 January 2004 (24.01.2004)

Date of receipt at the International Bureau: 02 March 2005 (02.03.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



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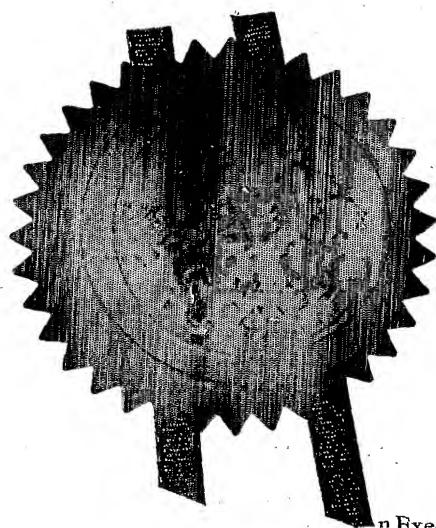
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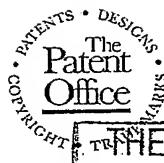
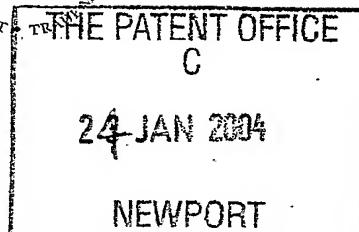
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GB0401561.6

By virtue of a direction under Section 30 of the patents Act 1977, the application is proceeding in the name of,

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[ADP No. 09023045001]

26 JAN 2004 E968057-2 DA2481
P01/7700 0.00-0401561.6 NONE

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3. Full name, address and postcode of the or of each applicant (underline all surnames)

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21/01/05

SECTION 3U (1977 ACT) APPLICATION FILED

Patents ADP number (if you know it)

3626174001

UK.

4. Title of the invention "Process for Particulate Material"

5. Name of your agent (if you have one)

Murgitroyd & Company

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to which all correspondence should be sent
(including the postcode)

Scotland House
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1198013

1198015

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Country

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Any other documents
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11.

I/We request the grant of a patent on the basis of this application.

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Date
23 January 2004

12. Name and daytime telephone number of person to contact in the United Kingdom

Mark Earnshaw

0141 307 8400

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1 Process for Particulate Material

2

3 The present invention relates to a process for
4 preparing a particulate material based on recycled
5 paper waste sludge containing cellulose fibres,
6 china clay and calcium carbonate, and a product
7 prepared therefrom.

8

9 There is an increasing industry in recycling waste
10 paper. Waste paper is increasingly collected
11 throughout the UK with intent to recycle it.
12 However, the term "waste paper" includes a myriad of
13 different types of paper-fibre materials, all of
14 which are provided by the public in the belief that
15 they are all 'recyclable' in the same way.

16

17 However, a number of the "waste papers" contain
18 materials that have been added during the original
19 paper production and conversion processes, and these
20 must be removed to provide a clean fibre material
21 suitable for re-use in a paper machine. Two main
22 materials that need to be removed from many types of

1 waste papers are china clay, also known as kaolin,
2 and calcium carbonate. These are added to certain
3 paper products to make the paper opaque and to
4 improve its printing quality. Other materials that
5 commonly need to be removed from waste papers are
6 staples, clips, glues, plastic coatings, etc.

7

8 Large separated materials from waste papers are
9 generally disposed of directly as "trash". These
10 include plastics, plastic coating materials and
11 metal clips, etc. Fine separated materials are
12 carried in an aqueous suspension to an effluent
13 treatment process where they are removed by gravity
14 sedimentation, and then further dewatered to 40-60%
15 solids for disposal. This creates a biological
16 sludge, (which can also be mixed with a primary
17 sludge before the dewatering stage).

18

19 These "paper waste sludge solids" are currently
20 simply deposited in landfill sites. However,
21 increasing environmental legislation requires this
22 depositing to be reduced.

23

24 Meanwhile, paper making processes generally also
25 create a paper-fibre based sludge waste product
26 which is also currently deposited in landfill sites.

27

28 A typical quantity of waste sludge from a paper
29 recycling and paper making facility can be 250
30 tonnes per day, representing a significant amount of
31 waste sludge material.

32

1 It is an object of the present invention to provide
2 a process for treating such waste material to form
3 an industrial useful product.

4

5 Thus, according to one aspect of the present
6 invention, there is provided a process for preparing
7 a particulate solid material comprising the steps
8 of:

9

10 obtaining a paper-fibre waste solid material having
11 a ratio of china clay, or equivalent, to chalk, or
12 equivalent, greater than a pre-determined minimum;

13

14 treating the material to reduce the moisture content
15 and form a granular material; and

16

17 calcining the granular material at a temperature of
18 approximately 1300°C or higher to provide a
19 particulate, 100% solids, material.

20

21 The paper-fibre based material can be provided by
22 the non-hazardous waste material arising from the
23 recycling of waste paper and tissue. Such material
24 is generally in the form of sludge, having a
25 moisture content of over 45%, and commonly over 55%
26 60%, or higher. Such sludge contains china clay,
27 calcium carbonate as well as the general cellulose
28 fibre content. Such material may also include
29 surplus biomass from biological effluent treatment
30 processes and water treatment processes which
31 produce sludge. Minor components may include non-
32 fibrous 'contraries' materials arising from waste

1 paper, including such items as polythene, plastics,
2 metal (in the form of wire, staples, paper clips).

3

4 The term china clay or equivalent includes any form
5 of hydrated aluminium silicate, including kandites,
6 kaolins and the like.

7

8 The term chalk or equivalent includes any form of
9 calcium carbonate, which includes the forms of
10 limestone.

11

12 The sludge is preferably dewatered so as to produce
13 a sludge having an increased solids content.

14

15 Analysis of the china clay:chalk ratio is preferably
16 carried out prior to the dewatering of the waste
17 material.

18

19 One method of analysis is termed 'acid extraction'.
20 A sample of sludge cake of known dry solids is
21 treated with 10% acetic acid solution to dissolve
22 calcium carbonate. The remaining solids are
23 filtered out, washed, dried at 105°C and weighed.
24 The loss in weight determines the calcium carbonate
25 content of the dry solids content of the sludge
26 cake. The remaining solids are further heated to
27 800°C to form an ash free from carbon which again is
28 weighed. The further loss in weight determines the
29 cellulose content of the sludge cake. The remaining
30 weight of ash determines the clay content. From
31 these calculated values the clay:chalk (calcium
32 carbonate) ratio is calculated.

1 By the acid extraction method, the pre-determined
2 minimum is approximately 0.2.

3
4 Another method of analysis is termed "ash/acid
5 extraction". For this, a weighed sample of dried
6 sludge cake is treated to 800°C to form an ash free
7 form carbon. The cooled sample is weighed and the
8 loss in weight determines the cellulose content plus
9 the carbon dioxide arising from the destruction of
10 the calcium carbonate. The cooled sample is then
11 treated with 10% acetic acid, filtered, washed,
12 dried and weighed. The further loss in weight
13 determines the calcium oxide content and the
14 remaining weight determines the clay content. From
15 these measured values the cellulose, calcium
16 carbonate and clay contents can be calculated.

17
18 By the ash/acid extraction method, the pre-
19 determined minimum is approximately 0.13.

20
21 Dewatering is a process well known in the art, as
22 are the process parameters for the pressing action.
23 Traditionally, a polyelectrolyte is added to a waste
24 material, which material can often have only a 7%
25 solids content, in order to agglutinate the very
26 fine waste material, commonly termed "fines", in the
27 process. The dewatering process increases the
28 solids content several fold, such as to a typical
29 solids content of 45%.

30
31 Where the china clay:chalk ratio is found to be less
32 than the pre-determined minimum in the waste

1 material, an embodiment of the present invention is
2 to add a conditioning material to the waste
3 material. The conditioning material is preferably
4 partly, substantially or wholly china clay, or at
5 least a china clay suspension, or another silicate
6 material having the same properties. A dispersing
7 agent could also be added to the conditioning material
8 in order to maintain the china clay or similar
9 material in a suspended form, in a liquid host such
10 as water.

11

12 It has been found that by varying the conditioning
13 material content in the waste material, the
14 dewatering process can result in a material having
15 less solids content than, for example 45%, such
16 content being even 22% or lower.

17

18 In an embodiment of the present invention, the
19 conditioning material is added to the waste material
20 even when the china clay:chalk ratio is greater than
21 the pre-determined minimum, in order to effect the
22 properties of the material as treated thereafter.

23

24 The dewatering process provides a sludge material
25 having a solids content generally in the range 22-
26 55%.

27

28 In another embodiment of the present invention, the
29 ratio of silica and aluminium to natural fillers in
30 the paper sludge is based on the addition of the
31 conditioning material which uses silica and/or
32 aluminium to promote fluxing of the paper sludge.

1 The treatment step can be provided by direct heat
2 contact, such as conduction. A heat transfer
3 material could be used such as steam, etc. in this
4 regard. The action of the heat treatment is to
5 partly or substantially 'dry' the paper-fibre based
6 material.

7
8 Such a heat treatment process could be carried out
9 with agitation, such as provided by a rotary
10 apparatus such as a rotary dryer. An inclined
11 rotary processor can reduce water content while
12 inducing a tumbling action against heated surfaces
13 at controlled temperatures. Preferably, any heat
14 treating apparatus allows for a wholly or
15 substantially continuous feed of starting material.
16 The process may involve recycling of material to
17 increase the range of particle sizes of granular
18 material produced.

19
20 The treatment step is preferably carried out at a
21 raised temperature, preferably between 60-80°C,
22 although not limited thereto.

23
24 The treatment step could also be carried out under
25 an inert atmosphere. Such an inert atmosphere could
26 be provided by displacing air with steam, either by
27 direct injection or by evaporated water. Heat
28 treating the material in an inert atmosphere reduces
29 the moisture content and forms the material into
30 rounded granules approximately 3mm-30mm in size.

1 The treatment step of the present invention could
2 also be carried out by operating on the paper-fibre
3 waste material in other ways, such as compression,
4 extrusion or the like, and/or a combination of such
5 processes. Extrusion through a die with apertures
6 produces lines of a material which either inherently
7 or by further processing produces an granulated
8 material.

9

10 In another embodiment of the present invention, the
11 granular material formed by the treatment step could
12 be further granulated, that is further processed in
13 a granulator or the like to better form a more
14 regular, generally spherical, shaped solids
15 material.

16

17 The material formed by the treatment step preferably
18 has a solids content in the range of approximately
19 50-90% solids. It has been found that the moisture
20 content of the so-formed granular material affects
21 the size of the particulate material formed in the
22 subsequent calcining step.

23

24 The calcining of the granular material is adapted to
25 reduce the moisture in the material to zero. The
26 particulate material being formed also becomes
27 porous, either partly or substantially, by the
28 burning of the cellulose content of the waste
29 material.

30

31 The calcining also fuses the granular material, so
32 as to provide solid pellets.

1 Preferably, the granular material is calcined with
2 agitation, such as provided by a rotary apparatus;
3 an example is a high temperature rotary furnace,
4 such as a tube. The rotary action serves to provide
5 a more even evaporation of moisture and burning of
6 cellulose.

7 Preferably, the calcining temperature is greater
8 than 1300°C, possibly approximately 1320°C or higher.
9

10 According to a second aspect of the present
11 invention, there is provided a particulate solid
12 material whenever prepared by a process as herein
13 described.

14 The particulate solid material preferably has a bulk
15 density of less than 900kg/m³, and generally in the
16 range 560 kg/m³ to 800 kg/m³. The particulate solid
17 material is preferably in the form of an aggregate.
18 Preferably, the aggregate has a mean particle size
19 of between 3-15mm. The mean particle size can be
20 favoured based on the moisture content of the
21 material made by the treatment step. The use of a
22 drier material provides a smaller mean particle
23 size, whereas the use of wetter material provides a
24 larger mean particle size.

25 The particulate solid material may be usable in a
26 number of industrial applications, including as a
27 light weight aggregate for making cementitious,
28 concrete or other building blocks, or as a
29

1 replacement or filler material in other building
2 applications. The material is also 'eco-friendly'.
3 Examples of the present invention will now be
4 described by way of example only.

5

6 Example 1

7

8 The starting material was provided from waste from
9 paper recycling in the form of raw separated solids
10 with water or sludge from biological or chemical
11 treatment of the separated solids. The starting
12 material included china clay, calcium carbonate,
13 cellulose fibres and a water content of
14 approximately 93%.

15

16 The material was analysed by the acid extraction
17 procedure to determine the ratio of clay:chalk and
18 to further determine the amount of conditioning
19 material to be added to bring the clay:chalk ratio
20 up to 0.2.

21

22 The conditioning material may include clay or chalk
23 and is prepared as a suspension in water at a
24 suitable concentration to facilitate pumping.

25

26 The material was fed to dewatering equipment with
27 conditioning material injected into the feed
28 pipeline. Dewatered material was collected from the
29 dewatering equipment with water content varying from
30 55% to 80%.

31

1 The dewatered material was fed at 10-15 kg/h into a
2 drier where heat transfer by contact with hot
3 surfaces evaporated water, formed the material into
4 rounded granules and provided an inert atmosphere.
5 The rounded granules were from 3-30mm in size, with
6 occasional granules up to 50mm in size, with a water
7 content of approximately 50%.

8

9 The dried material was fed at approximately 15 kg/h
10 into a calciner comprising a rotating tube lined
11 with refractory material and equipped with a propane
12 burner. The calciner was inclined so that material
13 passed counter current to the gas flow, passed
14 through a hot zone at 1300°C-1350°C before being
15 discharged into a receiving tray. The residence
16 time in the calciner was approximately 15-20 minutes
17 and the final processed material was hard to the
18 touch, light brown with a speckled surface in
19 appearance and produced a clear sound when dropped
20 onto a hard surface.

21

22 Example 2

23

24 The starting material was provided from waste from
25 paper recycling in the form of raw separated solids
26 with water or sludge from biological or chemical
27 treatment of the separated solids. The starting
28 material included china clay, calcium carbonate,
29 cellulose fibres and a water content of
30 approximately 93%.

31

1 The material was analysed by the acid extraction
2 procedure to determine the ratio of clay:chalk and
3 to further determine the amount of conditioning
4 material required to bring the clay:chalk ration to
5 >0.2.

6

7 The conditioning material may include clay or chalk
8 and in this example was presented as a filter cake
9 which was prepared as a suspension in water at a
10 suitable concentration to facilitate pumping.

11

12 The material was fed to dewatering equipment with
13 the conditioning material injected into the feed
14 pipeline. Dewatered material was collected with
15 water content varying from between 40% and 45%.

16

17 The dewatered material was fed to a granulating
18 press fitted with an extrusion plate thus producing
19 extruded particles of approximately 20mm diameter
20 having a cylindrical form.

21

22 The extruded particles were transferred to a
23 granulating table comprising an inclined surface
24 with a rim to retain material, and inclined at an
25 angle so that rotation of the table produced rounded
26 particles. Small quantities of water were sprayed
27 onto the particles to assist with rounding of them.
28 Undersized and fine particles were separated by
29 screening and were returned to the granulating press
30 to be reformed.

31

1 The rounded particles were fed at a rate up to
2 approximately 35 kg/h into a calciner comprising a
3 rotating tube lined with refractory material and
4 equipped with a gas burner. The calciner was
5 inclined so that material passed counter current to
6 the gas flow and passed through a hot zone before
7 being discharged into a receiving tray. The final
8 processed material was hard to the touch and light
9 brown with a speckled surface in appearance. The
10 bulk density of the product varied from 560 kg/m³ to
11 920 kg/m³ by adjusting the feed rate and speed of
12 rotation of the calciner.

13

14 The present invention provides an eco-friendly
15 method of using a significant waste product, that is
16 currently simply deposited in landfill sites. The
17 process produces a material which is usable in a
18 number of industrial applications, thereby not only
19 increasing the recyclability of waste papers, but
20 provided a beneficial product.

